

Effects of treatments on the properties of polyester based hemp composite

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The aim of present study is to enhance the properties of polyester based hemp composites by using different treatments. The effect of different treatments, such as alkali and benzoxylation (chemical treatments), and sodium bicarbonate (ecofriendly treatment), on water absorption, and mechanical & dynamic mechanical properties of hemp/polyester composites has been studied. The composites are prepared by hand lay-up technique using constant (15 wt. %) fibres content. Water absorption properties are investigated in terms of maximum water uptake, and sorption, diffusion & permeability coefficients. Dynamic mechanical properties, such as storage modulus (E'), glass transition temperature (T_g) & damping ($\tan\delta$), and mechanical properties such as tensile strength & modulus, flexural strength & modulus, and impact strength are also investigated. The results suggest a significant effect of chemical treatment in terms of increase in mechanical and dynamic mechanical properties, and decrease in water absorption properties. The benzoxylation treatment shows the better impact among all three chemical treatments.

Keywords: Dynamic mechanical properties, Hemp/polyester composite, Mechanical properties, Polyester, Water absorption properties

1 Introduction

Over recent past decades, natural fibres have obtained an increasing attention as a suitable alternative of synthetic fibres (mainly glass and carbon) for polymer based composites because of their excellent properties. These fibres offer low density & cost, high specific strength & modulus, and less wear & tear¹⁻⁵. In addition to this, their biodegradability makes them most attractive as compared to synthetic fibres⁶⁻⁷. Moreover, these fibres are recyclable and absorb CO₂ in the period of their growth⁸⁻⁹. On the other hand, these fibres also possess some drawbacks such as high moisture absorption, poor interaction with polymers, and low impact strength & durability¹⁰⁻¹⁵.

The fibres surface modification is commonly carried out to improve the adhesion between fibres and polymer matrix by various physical and chemical treatments, leading to increase in strength and decrease in moisture uptake of its composites^{16,17}. Physical treatments, such as plasma treatments, electron beam irradiation and corona treatment change the surface properties of the natural fibres, leading to enhancement in adhesion between fibres

and matrix without change in chemical composition. In addition, chemical treatments such as alkali, silane and benzoxylation not only increase the bonding between fibres and matrix but also change the chemical compositions of natural fibres¹⁶⁻¹⁸.

Dhanalakshmi *et al.*¹⁹ studied the effect of chemical treatments (alkali, permanganate, benzoyle and acrylate) on mechanical properties of areca fibre reinforced natural rubber composites which were prepared by heat press machine with various fibre loading (40%, 50%, 60% and 70%). It was reported that acrylate treated composites with 60% fibre loading has the highest values of flexural and impact strength amongst all composites. Mechanical properties are found to increase by alkali treatment of various natural fibres reinforced polymer composites such as alfa fibre reinforced polypropylene composite²⁰, coir fibre reinforced polymer composite²¹, roystonea regia fibre reinforced epoxy composite²² and sisal fibre reinforced epoxy composite²³. Wang *et al.*¹⁷ studied the effect of benzoyl chloride on properties of flax fibre low density polyethylene composite and obtained 6% and 33% improvement in tensile strength and water resistance properties respectively.

In present work, effect of alkali, benzoxylation and sodium bi carbonate treatments on the properties of hemp/polyester composites are reported. Alkali and

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benzoylation treatment are chemical treatments and have been used earlier by many researchers²⁴⁻²⁷. On the other hand, sodium bi carbonate is an ecofriendly treatment and has been used with sisal fibres. However, to the best of my knowledge no study has been carried out on eco friendly treatment of hemp fibre reinforced polyester composite. Therefore, a detailed study has been carried out on the effect of chemical treatments and ecofriendly treatment on the water absorption, mechanical and dynamic mechanical properties of hemp/polyester composite.

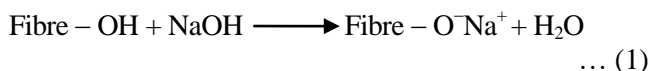
2 Materials and Methods

2.1 Materials

Hemp fibres were purchased from Uttarakhand Bamboo and Fibre Development Board, Dehradun, India. Unsaturated polyester resin with catalyst and accelerator were purchased from the local resource. The physical & mechanical properties and chemical composition of hemp fibre are provided in Table 1²⁸.

2.2 Alkali Treatment

Alkaline treatment is a commonly used chemical treatment of natural fibres which causes disturbance in hydrogen bonds, resulting in increase in surface roughness and hence enhancement in fibres-matrix adhesion. Reactions of NaOH with hemp fibre are given as follows:



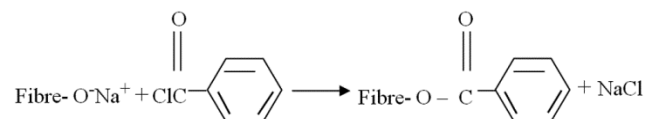
Alkali treatments were carried out using 5% NaOH concentration at 30 °C temperature, maintaining ML ratio at 1:15. The fibres were immersed in NaOH solution for 30 min, and then cleaned several times with distilled water followed by immersion in very dilute HCl in order to remove the NaOH adhering to

the surface of the fibres. Finally, the fibres were again washed several times with distilled water and then dried in a hot air oven at 70 °C for 24 h.

2.3 Benzoylation Treatment

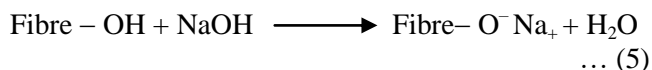
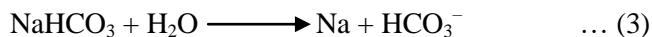
Benzoylation is the most frequently used chemical treatment which uses benzoyl chloride. The benzoyl chloride includes benzoyl ($\text{C}_6\text{H}_5\text{C}=\text{O}$) which is attributed to decrease in hydrophilic nature of natural fibres but it improves bonding with hydrophobic polymers matrix, thereby increasing the strength of composites.

The 5% NaOH pre-treated hemp fibres were immersed in 5% benzoyl chloride solution for 15 min at 30 °C. Subsequently, treated fibres were washed and dried, and then immersed in ethanol for 1 h to remove the adhering of benzoyl chloride. Finally, the treated fibres were washed with distilled water and dried in the hot air oven at 70 °C for 24 h. The reaction between the cellulosic $-\text{OH}$ group of fibre and benzoyl chloride is given as follows:



2.4 Sodium Bicarbonate Treatment

The extracted hemp fibres were washed several times by clean water and then dried at 30 °C for 48 h. The dried and clean hemp fibres were soaked in 5 % NaHCO_3 solution for 24 h at 30 °C, then washed with distilled water and dried in the hot air oven at 70 °C for 24 h. Reaction of sodium bicarbonate treatment on hemp fibre can explained as follows:



2.5 Fabrication of Composites

The treated hemp fibres were reinforced into matrix of unsaturated polyester resin to make the composites by hand lay-up technique followed by static compression. The curing of polyester resin was carried out at 30 °C by mixing one vol.% of ketone peroxide as catalyst and one vol.% of cobalt naphthenate as accelerator in order to make its polymer matrix. The mixture was stirred thoroughly to ensure a consistent mixing. A stainless steel mould having dimensions of 300 mm × 200 mm × 3 mm was used to make the composite laminate of 3 mm

Table 1 — Physical and mechanical properties, and chemical composition of hemp fibre

Properties	Values
Density, g/m ³	1.47
Diameter, µm	25-600
Elongation at break, %	2.0-4.0
Tensile strength, MPa	690
Young's modulus, GPa	70
Cellulose, %	70.2-74.4
Lignin, %	3.7-5.7
Microfibrillar angle, deg	2-6.2
Wax, %	0.8
Hemicellulose, %	17.9-22.4

thickness. Silicon spray was used to facilitate easy removal of the composite from the mould after curing. The cast of each composite was cured under a load of 50 kg for 24 h before it was removed from the mould. Specimens were cut in proper dimensions as per ASTM standard using a diamond cutter subjected to analysis of water absorption, mechanical and dynamic mechanical properties. The composites were manufactured using three types of treated hemp fibres (Table 2).

2.6 Test Methods

2.6.1 Water Absorption Behaviour

Water absorption behaviour of hemp composites was investigated according to ASTM D 570 standard. The specimens were immersed in the water at 30 °C to study the kinetics of water absorption behaviour. The samples were taken out periodically and weighed immediately after wiping off the water particles from the surface of the specimen using dry and clean cotton cloth. The weigh of the samples before and after absorption was measured using an electronic balance accurate to 10^{-4} g. The percentage of water absorption was calculated using the following equation :

$$\text{Water absorption (\%)} = \frac{w_2 - w_1}{w_1} \times 100 \quad \dots (6)$$

where w_1 is the weight before soaking into water (g); and w_2 , the weight after soaking into water (g).

At several periods of time, the percentage of water absorption was calculated and then plotted against square root of immersion time to calculate the diffusion coefficient. Diffusion coefficient was calculated from the slope of curve between percentage of water absorption and square root of immersion time using the following equation²⁹:

$$\text{Diffusion coefficient (D)} = \pi \left(\frac{t^2 m^2}{16W_\infty^2} \right) \quad \dots (7)$$

Table 2 — Notations of untreated and treated hemp composites

Code	Composites	Wt % (hemp/polyester)	Treatment concentration %
HC	Untreated hemp/ polyester	H15/P85	0
HCT1	NaHCO ₃ treated hemp/ polyester	H15/P85	5
HCT2	NaOH treated hemp/ polyester	H15/P85	5
HCT3	benzoylation treated hemp/ polyester	H15/P85	5

where m is the slope of linear portion of the sorption curve; and t , the initial sample thickness in (mm).

In addition, sorption coefficients that are related to the equilibrium sorption was calculated as follows²⁹:

$$\text{Sorption coefficient } S = W_\infty / W_t \quad \dots (8)$$

where W_∞ and W_t are the percentage of water uptake at saturation time and at time t .

The permeability coefficient that shows the net effect of sorption and diffusion coefficient was calculated as follows²⁹:

$$\text{Permeability coefficient (P)} = D \times S \quad \dots (9)$$

2.6.2 Tensile Test

Tensile test of the composite samples was performed on Tinius Olsen H 10 K-L (Bi-axial testing machine) with a crosshead speed of 2 mm/min. Tests were conducted as per ASTM D 638 with dimension of 165 mm × 20 mm × 3 mm. Five specimens of each composite were tested and their average values and standard deviations were reported.

2.6.3 Flexural Test

Flexural test of the composite samples was carried out using a three point bending test on Tinius Olsen H10 K-L (Bi-axial testing machine). The standard ASTM D790 was used for the flexural test with dimensions of 80 mm × 12.7 mm × 3 mm as per ASTM D790. The flexural test was also carried out at crosshead speed of 2 mm/min. Flexural strength and flexural modulus were calculated using following equations :

$$\text{Flexural strength} = \frac{3FL}{2bd^2} \quad \dots (10)$$

$$\text{Flexural modulus} = \frac{mL^3}{4bd^3} \quad \dots (11)$$

where F is the ultimate failure load (N); L , the span length (mm); b and d , the width and thickness of specimen in (mm) respectively; and m , the slope of the tangent to initial line portion of the load-displacement curve. Five specimens of each composite were tested and their average values and standard deviations were reported.

2.6.4 Impact Test

Izod Impact test with notch of the composite samples was performed on Tinius Olsen Impact 104 machine. The samples for the impact test were prepared in dimensions of 65 mm × 12.7 mm × 3 mm

and 2.5 mm notch thickness as per ASTM D 256. Five specimens of each composite were tested and their average values and standard deviations were reported.

2.6.5 Dynamic Mechanical Analysis

The dynamic mechanical properties of hemp composites were studied using the dynamic mechanical analyzer (Seiko instruments DMA 6100). The dynamic mechanical properties were carried out using 3 point bending test as a function of temperature. The laminates were cut into samples with dimensions of 50 mm × 13 mm × 3 mm as per ASTM D 5023. Experiments were carried out at 5 Hz frequency within temperature range of 30°–200 °C in order to analyze E' , $Tan \delta$ and T_g .

3 Results and Discussion

3.1 Water Absorption Behaviour

The percentage of water absorption has been plotted against the square root of time for untreated and treated hemp composites as shown in Fig. 1. It could be observed that the initial rate of water uptake is linear for all the composites but after extending in immersion time, water absorption slows down and then goes to saturation stages. Hence, for all hemp composites, behaviour of water absorption could be considered as Fickian diffusion process. It is observed that all three treatments show significantly decreased water absorption of hemp composite. The maximum value of % water absorption at saturation stage is found for HC (4.59) followed by HCT1 (4.17), HCT2 (3.84) and HCT3 (3.40). The maximum water absorption of HC is because of hydrophilic nature of hemp fibres due to the presence of hydroxyl groups²⁹.

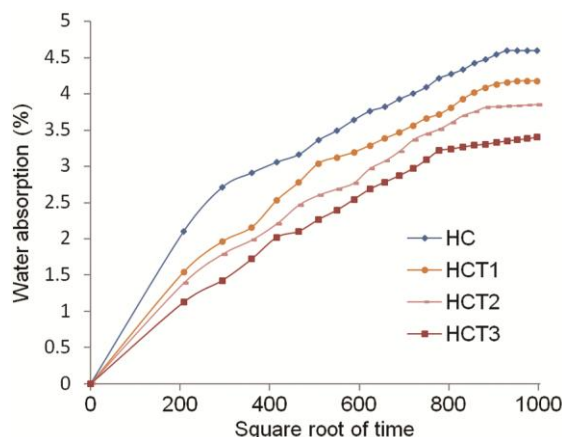


Fig. 1 — Water absorption characteristics of untreated and treated hemp composit

Alkali treated hemp composite HCT2 has 16% and 8% lower water absorption than those of HC and HCT1 respectively. The reduction in water absorption by alkali treatment is already reported^{30, 31}. Alkali treatment decreases the water absorption due to removal of hemicellulose and lignin from the surface of the fibres, thereby increasing the surface roughness of fibres which results in increased interfacial adhesion between fibres and matrix²⁹. The composite HCT3 has the lowest percentage of water uptake among all composites due to further action of benzoyl chloride on alkali treated hemp fibres, leading to increase in bonding between fibres and matrix and minimum number of micro voids.

Furthermore, diffusion, sorption and permeability coefficients are also investigated for treated and untreated hemp composites, and these results are summered in Table 3. Diffusion coefficient shows the ability of diffusion of water molecules into micro voids of the composites, and permeability coefficient shows the net effect of sorption and diffusion coefficient. Both coefficients are dependent on percentage of water uptake. Therefore, diffusion and permeability coefficients show linear relationship with % water absorption of the composites. On the other hand, sorption coefficient shows a reverse trend because it is a ratio of percentage of water absorption at saturation stage and the % of water absorption at time t ; where t is the final linear square root of immersion time (207.84). It means sorption coefficient is inversely proportional to percentage of water absorption at linear time t . At the end of linear time t , percentage of water absorption follows the order: HC > HCT1 > HCT2 > HCT3 (Fig. 1). Hence, sorption coefficients show the reverse trend of diffusion and permeability coefficients.

Moreover, among all treated and untreated hemp composites, the lowest value of diffusion coefficient is observed for HCT3, followed by HCT2, HCT1 and HC. The lowest value of diffusion coefficient of

Table 3 — Sorption, diffusion and permeability coefficient of untreated and treated hemp composites

Composite	% water uptake at saturation stage	Sorption coefficient (S)	Diffusion coefficient (D) × 10 ⁻⁶ mm ² /s	Permeability coefficient (P) × 10 ⁻⁶ mm ² /s
HC	4.59	2.17	8.557	1.870
HCT1	4.17	2.70	5.575	1.509
HCT2	3.84	2.78	5.279	1.469
HCT3	3.40	3.01	4.483	1.353

HCT3 is attributed to benzoilation treatment. The benzoilation treatment provides strong fibres-matrix adhesion, leading to minimum voids which results in higher resistance to diffusion of water molecules into these voids. Permeability coefficient follows the trend same as that of diffusion coefficient. However, the higher value of sorption coefficient is observed for HCT3 followed by HCT2, HCT1 and HC.

3.2 Tensile Properties

Tensile strength and tensile modulus of untreated and treated hemp composites are plotted in Fig. 2 (a). It is observed that all treated composites have the higher values of tensile strength and tensile modulus than that of untreated hemp composites. In addition to this, it can be observed that HCT3 has the highest

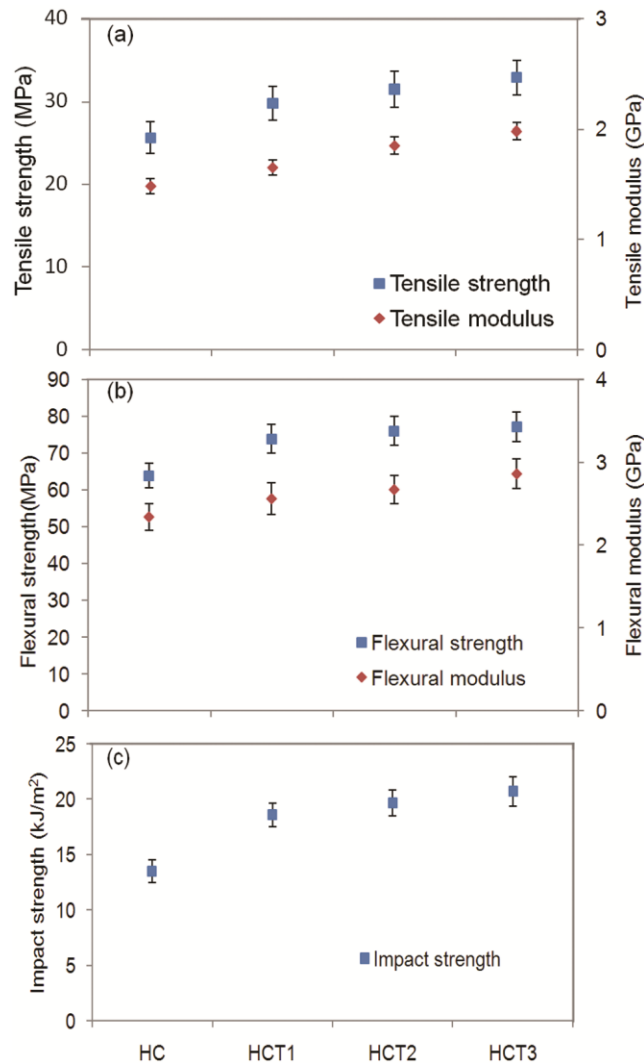


Fig. 2 — Mechanical properties of untreated and treated hemp composites (a) tensile strength and modulus, (b) flexural strength and modulus and (c) impact strength

values of tensile strength (32.85 MPa) and tensile modulus (1.98 GPa) among all composites; due to effect of benzoilation treatment. Increase in tensile properties due to benzoilation treatment is already studied by Wang *et al.*¹⁷. After HCT3, the maximum values of tensile strength and tensile modulus follow the order: HCT2 > HCT1 > HC. The tensile strength and tensile modulus of HCT2 are observed 31.45 MPa and 1.85 GPa respectively which are 23% and 25 % respectively higher than that of untreated hemp composite HC. A similar type of results as improvement in tensile properties due to alkali treatment has already been reported^{32, 33}. Sodium bicarbonate treated hemp composite HCT1 has 16% and 11% higher values of tensile strength and tensile modulus than that of HC; as the action of sodium bicarbonate leads to increment in fibre-matrix adhesion which provides uniform stress transfer. The increase in tensile properties of present hemp composite due to sodium bicarbonate treatment is in good agreement with the findings of Fiore *et al.*³⁴. This is strictly due to the removal of hemicellulose, and partial removal of lignin leads to diameter reduction, thereby increasing the aspect ratio L/d ³⁴. The increase in aspect ratio provides the larger area of fibres for adhesion with polymers.

3.3 Flexural Properties

Figure 2(b) shows the flexural strength and flexural modulus of untreated and treated hemp composites. A similar trend like tensile test is noticed for flexural tests in terms of strength and modulus. It is observed that all treated composites have noticeable improvement in values of flexural strength and flexural modulus than untreated hemp composites, which shows a positive effect of treatments. The highest values of flexural strength (77.15 MPa) and flexural modulus (2.86 GPa) are obtained for HCT3; 21% and 22% higher than that of HC. After HCT3, the maximum values of flexural strength and flexural modulus follow the same order of tensile test, as HCT2 > HCT1 > HC. The alkali-treated hemp composite (HCT2) has 19% and 14% higher values of flexural strength and flexural modulus respectively than that of HC. The increase in flexural properties due to alkali treatment is already reported by Ray *et al.*³⁵ and Prasad *et al.*³⁶. Furthermore, the sodium bicarbonate treated hemp composite HCT1 has the 16% and 9% higher flexural strength and flexural modulus respectively than that of HC. The increase in flexural properties due to sodium

bicarbonate treatment has already been reported by Fiore *et al.*³⁴.

3.3 Impact Properties

Impact strength of untreated and treated hemp composites is plotted in Fig. 2(c). Similar trend like those of tensile and flexural tests is also found in impact test. In case of impact test, it is also observed that all treated composites have higher values of impact strength than that of untreated hemp composite which shows a positive effect of treatments in terms of increase in impact property. The highest value of impact strength (20.71 kJ/m²) is observed for HCT3, which is 53% higher than that of HC. The alkali-treated hemp composite (HCT2) has 46% higher value of impact strength than that of HC. The increase in impact strength due to alkali treatment has already been reported^{30,36}. The sodium bicarbonate treated hemp composite (HCT1) has 38 % higher value of impact strength than that of untreated hemp composite HC.

3.4 Storage Modulus

Storage modulus can be defined as the maximum energy stored by the polymer composites during one

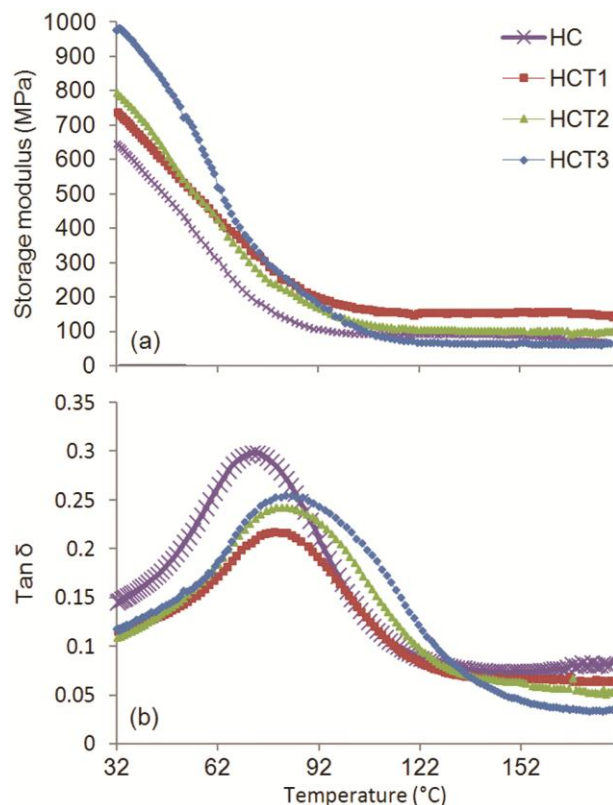


Fig. 3 — Variation in (a) storage modulus and (b) damping with temperature of untreated and treated hemp composites

cycle of oscillation. Figure 3(a) shows the variation in storage modulus of untreated and treated hemp composites as a function of temperature at 5 Hz frequency. It is interesting to note that all treated composites have higher values of storage modulus than untreated composites in glassy region. The maximum value of storage modulus is found for HCT3 followed by HCT2, HCT1 and HC (Table 4). The storage modulus of all the composites are found to decrease as the temperature increases which is due to loss in stiffness of fibres^{2, 7, 37,38}. In transition region, there is a gradual fall in the value of E' when temperature is increased [Fig. 3(a)]. This may be due to the increase in molecular mobility of polymer chain above the glass transition temperature. In rubbery region, the highest value of storage modulus follows the same order as in glassy region. This may be due to reinforcement of stiffer fibres because of treatments.

3.5 Damping

Damping or $Tan \delta$ is the ratio of loss modulus and storage modulus. It shows the impact properties of composite material and depends upon fibre-matrix adhesion, and strength and stiffness of fibres. Weak fibre-matrix adhesion shows the higher value of $Tan \delta$ and vice-versa. The effect of damping on hemp composites as a function of temperature is shown in Fig. 3(b). The highest peak of $Tan \delta$ is found for HC followed by HCT1, HCT2 and HCT3 (Table 4). The lowest value of $Tan \delta$ peak is seen for HCT3; which shows lower damping property and good load bearing capacity due to strong adhesion between fibres and polymer matrix. The maximum shifting of $Tan \delta$ curve towards right side is shown by HCT3 followed by HCT2, HCT1 and HC. The higher shifting of $Tan \delta$ curve towards right side presents the higher values of T_g of the composites. The values of $Tan \delta$ peaks and corresponding T_g are given in Table 4.

Table 4 — Values of peak heights and T_g from tan delta curve			
Composite	Peak height		T_g , °C (from $Tan \delta$ curve)
	Storage modulus curve, MPa	$Tan \delta$ curve	
HC	681	0.298	73.67
HCT1	803	0.216	77.48
HCT2	884	0.242	82.51
HCT3	981	0.254	87.18

4 Conclusion

4.1 All treated hemp composites show the better performance than untreated hemp composite.

4.2 The best mechanical properties is shown by benzoylation treated hemp composite HCT3 followed by HCT2, HCT1 and HC.

4.3 Dynamic mechanical properties such as storage modulus and glass transition temperature are found the maximum by HCT3.

4.4 The maximum water absorption resistance is shown by HCT3 followed by HCT2, HCT1 and HC.

4.5 Benzoylation treated hemp composite shows the best performance among all treated composites.

4.6 The ecofriendly chemical treatment such as sodium bi carbonate (baking soda) shows a significant improvement in properties of hemp composite.

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